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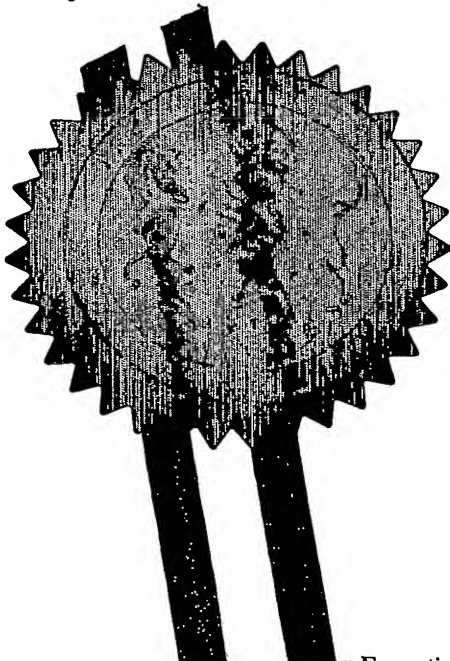
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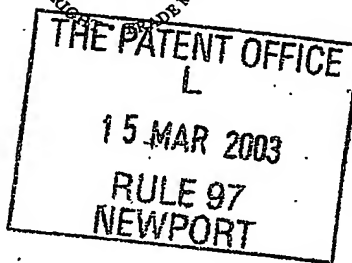


1/77

18MAR03 E792994-1 C12133  
P01/7700 0.00-0306097.7

# Request for grant of a patent

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565

2. Patent application number

(The Patent Office will fill in this part)

0306097.7

15 MAR 2003

3. Full name, address and postcode of the or of each applicant (underline all surnames)

ELAM-T LIMITED  
103 BOROUGH ROAD  
LONDON.  
SE1 OAA

Patents ADP number (if you know it)

8105280001

If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention

ELECTROLUMINESCENT COMPLEXES

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

COHEN, ALAN NICOL  
2 GROVE PLACE  
TATSFIELD  
Nr. WESTERHAM  
KENT  
TN16 2BB

Patents ADP number (if you know it)

6963557001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
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Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

YES

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Description 12

Claim(s) 4

Abstract 1

Drawing(s) 11 + 11

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Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*)

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I/We request the grant of a patent on the basis of this application.

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A. N. Cohen

01959 577172

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## Electroluminescent Complexes

The present invention relates to electroluminescent materials and to electroluminescent devices.

5

Materials which emit light when an electric current is passed through them are well known and used in a wide range of display applications. Liquid crystal devices and devices which are based on inorganic semiconductor systems are widely used, however these suffer from the disadvantages of high energy consumption, high cost of manufacture, low quantum efficiency and the inability to make flat panel displays.

10

Organic polymers have been proposed as useful in electroluminescent devices, but it is not possible to obtain pure colours, they are expensive to make and have a relatively low efficiency.

15

Another compound which has been proposed is aluminium quinolate, but this requires dopants to be used to obtain a range of colours and has a relatively low efficiency.

20

Patent application WO98/58037 describes a range of lanthanide and transition metal complexes which can be used in electroluminescent devices which have improved properties and give better results. Patent Applications PCT/GB98/01773, PCT/GB99/03619, PCT/GB99/04030, PCT/GB99/04024, PCT/GB99/04028, PCT/GB00/00268 describe electroluminescent complexes, structures and devices using rare earth chelates.

25

US Patent 5128587 discloses an electroluminescent device which consists of an organometallic complex of rare earth elements of the lanthanide series sandwiched between a transparent electrode of high work function and a second electrode of low work function with a hole conducting layer interposed between the

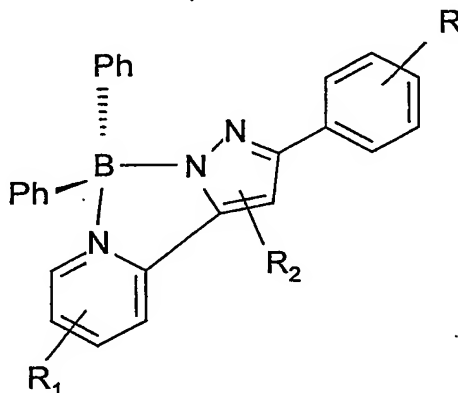
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electroluminescent layer and the transparent high work function electrode and an electron conducting layer interposed between the electroluminescent layer and the electron injecting low work function anode. The hole conducting layer and the electron conducting layer are required to improve the working and the efficiency of the device. The hole transporting layer serves to transport holes and to block the electrons, thus preventing electrons from moving into the electrode without recombining with holes. The recombination of carriers therefore mainly takes place in the emitter layer.

US Patents 6,287,713 and 6,368,731 the contents of which are incorporated by reference disclose electroluminescent compounds which are complexes of boron with 8-aminoquinolate derivatives.

We have now invented novel electroluminescent boron complexes.

According to the invention there is provided a boron complex of formula



(I)

where Ph is an unsubstituted or substituted phenyl group where the substituents can be the same or different and are selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring

structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups;  $R$ ,  $R_1$  and  $R_2$  can be hydrogen or substituted or unsubstituted hydrocarbyl groups, such as substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorine, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups or nitrile.

Examples of  $R$  and/or  $R_1$  and/or  $R_2$  and/or  $R_3$  include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and unsubstituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

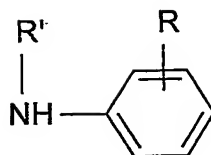
The invention also provides an electroluminescent device which comprises a first electrode, a layer of an electroluminescent material and a second electrode in which the electroluminescent material is a complex of formula (I).

The first electrode can function as the cathode and the second electrode can function as the anode and preferably there is a layer of a hole transporting material between the anode and the layer of the electroluminescent compound.

The hole transporting material can be any of the hole transporting materials used in electroluminescent devices.

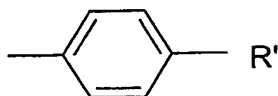
The hole transporting material can be an amine complex such as poly(vinylcarbazole),  $N, N'$ -diphenyl- $N, N'$ -bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD), an unsubstituted or substituted polymer of an amino substituted aromatic compound, a polyaniline, substituted polyanilines, polythiophenes, substituted polythiophenes, polysilanes etc. Examples of polyanilines are polymers of

- 4 -



(II)

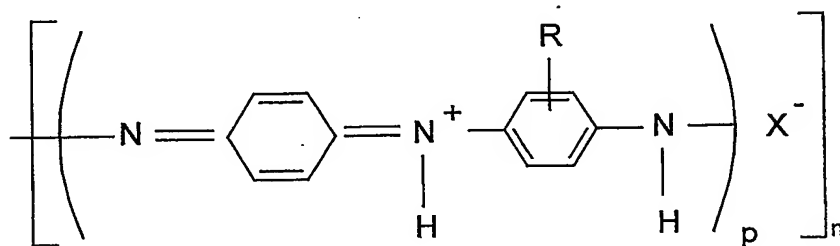
where R is in the ortho - or meta-position and is hydrogen, C1-18 alkyl, C1-6 alkoxy, amino, chloro, bromo, hydroxy or the group



5

where R is alkyl or aryl and R' is hydrogen, C1-6 alkyl or aryl with at least one other monomer of formula I above.

- 10 Or the hole transporting material can be a polyaniline, polyanilines which can be used in the present invention have the general formula



(III)

- 15 where p is from 1 to 10 and n is from 1 to 20, R is as defined above and X is an anion, preferably selected from Cl, Br, SO<sub>4</sub>, BF<sub>4</sub>, PF<sub>6</sub>, H<sub>2</sub>PO<sub>3</sub>, H<sub>2</sub>PO<sub>4</sub>, arylsulphonate, arenedicarboxylate, polystyrenesulphonate, polyacrylate alkylsulphonate, vinylsulphonate, vinylbenzene sulphonate, cellulose sulphonate, camphor sulphonates, cellulose sulphate or a perfluorinated polyanion.

Examples of arylsulphonates are p-toluenesulphonate, benzenesulphonate, 9,10-anthraquinone-sulphonate and anthracenesulphonate, an example of an arenedicarboxylate is phthalate and an example of arenecarboxylate is benzoate.

- 5 We have found that protonated polymers of the unsubstituted or substituted polymer of an amino substituted aromatic compound such as a polyaniline are difficult to evaporate or cannot be evaporated, however we have surprisingly found that if the unsubstituted or substituted polymer of an amino substituted aromatic compound is deprotonated then it can be easily evaporated i.e. the polymer is evaporable.

10

Preferably evaporable deprotonated polymers of unsubstituted or substituted polymer of an amino substituted aromatic compound are used. The de-protonated unsubstituted or substituted polymer of an amino substituted aromatic compound can be formed by deprotonating the polymer by treatment with an alkali such as ammonium hydroxide or an alkali metal hydroxide such as sodium hydroxide or potassium hydroxide.

15

The degree of protonation can be controlled by forming a protonated polyaniline and de-protonating. Methods of preparing polyanilines are described in the article by A. G. MacDiarmid and A. F. Epstein, Faraday Discussions, Chem Soc.88 P319 1989.

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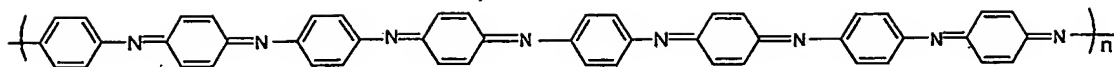
The conductivity of the polyaniline is dependant on the degree of protonation with the maximum conductivity being when the degree of protonation is between 40 and 60% e.g. about 50% for example.

25

Preferably the polymer is substantially fully deprotonated.

A polyaniline can be formed of octamer units i.e. p is four e.g.





The polyanilines can have conductivities of the order of  $1 \times 10^{-1}$  Siemen  $\text{cm}^{-1}$  or higher.

5

The aromatic rings can be unsubstituted or substituted e.g. by a C1 to 20 alkyl group such as ethyl.

10

The polyaniline can be a copolymer of aniline and preferred copolymers are the copolymers of aniline with o-anisidine, m-sulphanilic acid or o-aminophenol, or o-toluidine with o-aminophenol, o-ethylaniline, o-phenylene diamine or with amino anthracenes, o-toluidine, o-ethylaniline, m-toluidine, m-ethylaniline etc.

15

Other polymers of an amino substituted aromatic compound which can be used include substituted or unsubstituted polyaminonaphthalenes, polyaminoanthracenes, polyaminophenanthrenes, etc. and polymers of any other condensed polyaromatic compound. Polyaminoanthracenes and methods of making them are disclosed in US Patent 6,153,726. The aromatic rings can be unsubstituted or substituted e.g. by a group R as defined above.

20

Other hole transporting materials are conjugated polymer and the conjugated polymers which can be used can be any of the conjugated polymers disclosed or referred to in US 5807627, PCT/WO90/13148 and PCT/WO92/03490.

25

The preferred conjugated polymers are poly (p-phenylenevinylene)-PPV and copolymers including PPV. Other preferred polymers are poly(2,5 dialkoxyphenylene vinylene) such as poly (2-methoxy-5-(2-methoxypentyloxy)-1,4-phenylene vinylene), poly(2-methoxypentyloxy)-1,4-phenylenevinylene), poly(2-methoxy-5-(2-dodecyloxy-1,4-phenylenevinylene) and other poly(2,5 dialkoxyphenylenevinylens)

with at least one of the alkoxy groups being a long chain solubilising alkoxy group, poly fluorenes and oligofluorenes, polyphenylenes and oligophenylenes, polyanthracenes and oligo anthracenes, ploythiophenes and oligothiophenes.

- 5 In PPV the phenylene ring may optionally carry one or more substituents e.g. each independently selected from alkyl, preferably methyl, alkoxy, preferably methoxy or ethoxy.

- 10 In poly(fluorene), the fluorene ring may optionally carry one or more substituents e.g. each independently selected from alkyl, preferably methyl, alkoxy, preferably methoxy or ethoxy.

- Any poly(arylenevinylene) including substituted derivatives thereof can be used and the phenylene ring in poly(p-phenylenevinylene) may be replaced by a fused ring system such as anthracene or naphthylene ring and the number of vinylene groups in each polyphenylenevinylene moiety can be increased e.g. up to 7 or higher.
- 15

- The conjugated polymers can be made by the methods disclosed in US 5807627, PCT/WO90/13148 and PCT/WO92/03490.

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The thickness of the hole transporting layer is preferably 20nm to 200nm.

- The polymers of an amino substituted aromatic compound such as polyanilines referred to above can also be used as buffer layers with or in conjunction with other hole transporting materials e.g. between the anode and the hole transporting layer. Other buffer layers can be formed of phthalocyanines such as copper phthalocyanine.
- 25

- The structural formulae of some other hole transporting materials are shown in Figures 3 to 7 of the drawings, where  $R_1$ ,  $R_2$  and  $R_3$  can be the same or different and are selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups
- 30

such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups;  $R_1$ ,  $R_2$  and  $R_3$  can also form substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer e.g. styrene. X is Se, S or O, Y can be hydrogen, substituted or unsubstituted hydrocarbyl groups, such as substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorine, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups or nitrile.

10

Examples of  $R_1$  and/or  $R_2$  and/or  $R_3$  include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and unsubstituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

15

Optionally there is a layer of an electron injecting material between the cathode and the electroluminescent material layer, the electron injecting material is a material which will transport electrons when an electric current is passed through electron injecting materials include a metal complex such as a metal quinolate e.g. an aluminium quinolate, lithium quinolate,  $Mx(DBM)_n$  where  $Mx$  is a metal and DBM is dibenzoyl methane and  $n$  is the valency of  $Mx$  e.g.  $Mx$  is aluminium or chromium.

20

In place of the DBM moiety there can be a Schiff base.

25

The electron injecting material can also be a cyano anthracene such as 9,10 dicyano anthracene, cyano substituted aromatic compounds, tetracyanoquinodimethane a polystyrene sulphonate or a compound with the structural formulae shown in figures 1 or 2 of the drawings in which the phenyl rings can be substituted with substituents R as defined above. Instead of being a separate layer the electron injecting material can be mixed with the electroluminescent material and co-deposited with it.

30

Optionally the hole transporting material can be mixed with the electroluminescent material and co-deposited with it and the electron injecting materials and electroluminescent materials can be mixed.

- 5 The hole transporting materials, the electroluminescent material and the electron injecting materials can be mixed together to form one layer, which simplifies the construction.

10 The anode is preferably a transparent substrate such as a conductive glass or plastic material which acts as the anode, preferred substrates are conductive glasses such as indium tin oxide coated glass, but any glass which is conductive or has a conductive layer such as a metal or conductive polymer can be used. Conductive polymers and conductive polymer coated glass or plastics materials can also be used as the substrate.

15

The cathode is preferably a low work function metal e.g. aluminium, barium, rare earth metals, transition metals, calcium, lithium, magnesium and alloys thereof such as silver/magnesium alloys, rare earth metal alloys etc., aluminium is a preferred metal. A metal fluoride such as an alkali metal e.g. lithium fluoride, rare earth metal  
20 fluoride can be used as the second electrode for example by having a metal fluoride layer formed on a metal.

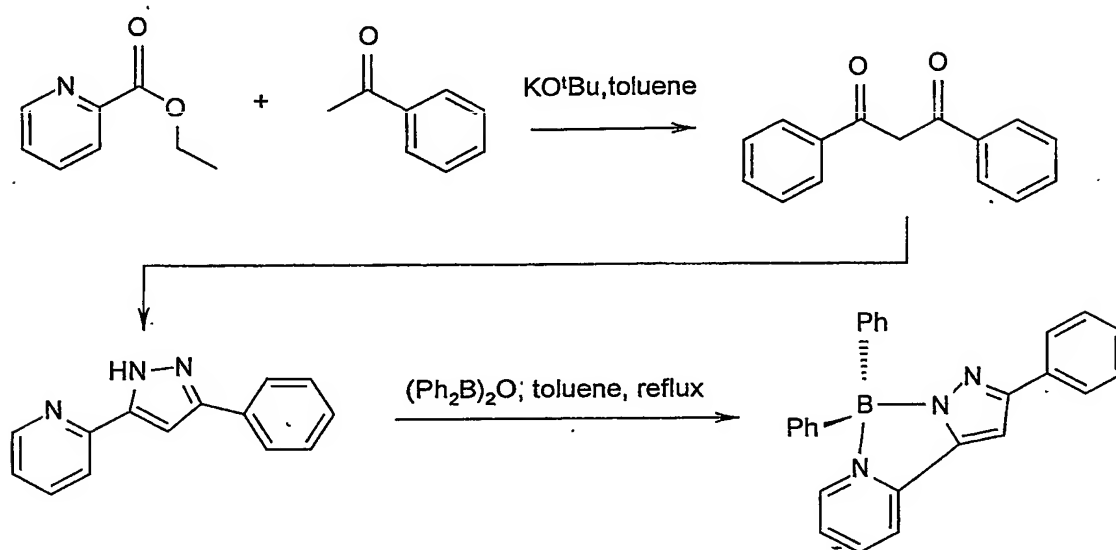
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The boron complexes of the present invention include blue emitting electroluminescent materials.

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# Example 1

## Preparation of 2-(2-diphenylboranyl-5-phenylpyrazol-3-yl)pyridine



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Acetophenone (11.6 mL, 99 mmol), was added to a suspension of potassium tert-butoxide (12.34g 110 mmol) in toluene (150 mL) and stirred under argon. The flask was then charged with ethyl picolinate (13.4 mL, 99 mmol) and the mixture stirred overnight. The toluene was removed by rotary evaporation and 200 mL of both diethylether and deionised water added. With stirring the mixture was acidified with dilute HCl until the ether layer was dark orange ~ pH 7). The ether layer was separated, washed with deionised water (3 x 100 mL), dried over magnesium sulfate and filtered. The solvent was removed and the solid recrystallised from hot ethanol to yield 1-phenyl-3-pyridin-2-ylpropane-1,3-dione (1) as a pale yellow crystalline solid (15.3 g, 69%).

A 250 mL flask was charged with 1 (10g 44 mmol), a magnetic follower and 150 mL of ethanol. Hydrazine monohydrate (2.15 mL, 44 mmol) was added and the mixture refluxed overnight. On cooling the ethanol was removed and the residue recrystallised from methanol to yield 2-(5-phenylpyrazol-3-yl)pyridine (2) as a pale

20

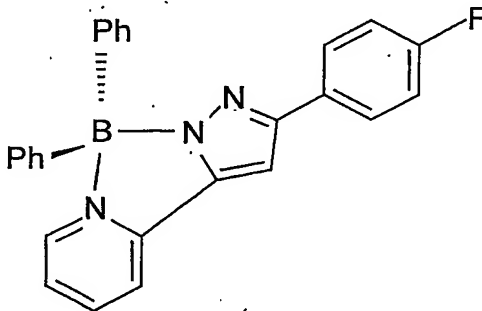
yellow crystalline solid (9.5 g, 97 %)

A 150 mL flask was charged with diphenylborinic anhydride (1.00 & 2.9 mmol), 2(1.28 g, 5.8 mmol) and 100 mL of toluene. The mixture was refluxed until all traces of the anhydride had dissolved and then refluxed for a further 2h. On cooling, white crystals were formed. These were collected by filtration and dried under vacuum to give 2.15g (96%) of 2-(2-diphenylboranyl-5-phenylpyrazol-3-yl)pyridine 3. DSC:- Mpt. 261.4- 265.2° C (Recryst. 173.3 °C).

The properties of this complex are shown in table 1 below and its emission spectrum and UV absorbance are shown in figs. 8 and 9.

#### Example 2

##### Preparation of 2-(2-diphenylboranyl-5-(4-fluorophenyl)pyrazol-3-yl)pyridine



2-(2-diphenylboranyl-5-(4-fluorophenyl)pyrazol-3-yl)pyridine was prepared by the method of example 1 with acetophenone being replaced by 4'-fluoroacetophenone. The diketone was prepared in ~70% yield and the free ligand and boron compounds were formed in 90+% yields. DSC; Mpt. 263.7- 266.6°C (Recryst.196.4°C).

The properties of this complex are shown in table 1 below and its emission spectrum and UV absorbance are shown in figs. 10 and 11.

Table 1

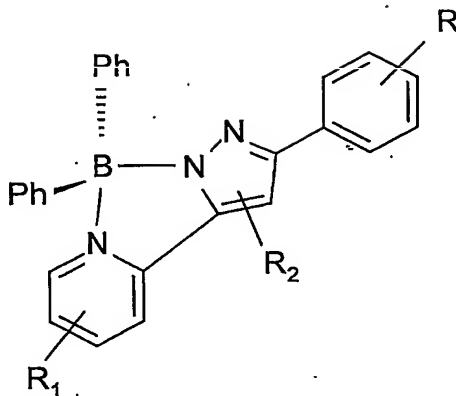
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Compound	PL Efficiency $\text{Cdm}^{-2}\mu\text{w}^{-1}$	Peak Wavelength nm	CIE Coord. x;y	M.P.°C
1	0.071	~455	0.17;0.16	263-267
2	0.058	~450	0.16;0.15	261-265

## Claims

1. A boron complex of formula

5



(I)

10 where Ph is an unsubstituted or substituted phenyl group where the substituents can be the same or different and are selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups; R, R<sub>1</sub> and R<sub>2</sub> can be hydrogen or substituted or unsubstituted  
15 hydrocarbyl groups, such as substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorine, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups or nitrile.

20 2. A complex as claimed in claim 1 in which R and/or R<sub>1</sub> and/or R<sub>2</sub> are aliphatic, aromatic or heterocyclic alkoxy, aryloxy and carboxy groups, substituted and substituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.



3. An electroluminescent device which comprises a first electrode, a layer of an electroluminescent material as claimed in any one of claims 1 or 2 and a second electrode.
- 5 4. An electroluminescent device as claimed in claim 3 in which there is a layer of a hole transmitting material between the first electrode and the electroluminescent layer.
- 10 5. An electroluminescent device as claimed in claim 3 in which the hole transmitting material is an aromatic amine complex.
6. An electroluminescent device as claimed in claim 3 in which the hole transmitting material is polyaromatic amine complex.
- 15 7. An electroluminescent device as claimed in claim 3 in which the hole transmitting material is a film of a polymer selected from poly(vinylcarbazole), N,N'-diphenyl-N,N'-bis (3-methylphenyl) -1,1' -biphenyl -4,4'-diamine (TPD), CBP, polyaniline, substituted polyanilines, polythiophenes, substituted polythiophenes, polysilanes and substituted polysilanes.
- 20 8. An electroluminescent device as claimed in claim 4 in which the hole transmitting material is a film of a compound of formula (II) or (III) herein or as in figures 3 to 7 of the drawings.
- 25 9. An electroluminescent device as claimed in claim 3 in which the hole transmitting material is a copolymer of aniline, a copolymer of aniline with o-anisidine, m-sulphanilic acid or o-aminophenol, or o-toluidine with o-aminophenol, o-ethylaniline, o-phenylene diamine or with an amino anthracene, 0-toluidine, o-ethylaniline, m-toluidine, m-ethylaniline etc.

10. An electroluminescent device as claimed in claim 3 in which the hole transmitting material is a conjugated polymer.

5 11. An electroluminescent device as claimed in claim 10 in which the conjugated polymer is selected from poly (p-phenylenevinylene)-PPV and copolymers including PPV, poly(2,5 dialkoxyphenylene vinylene), poly (2-methoxy-5-(2-methoxypentyloxy-1,4-phenylene vinylene), poly(2-methoxypentyloxy)-1,4-phenylenevinylene), poly(2-methoxy-5-(2-dodecyloxy-1,4-phenylenevinylene) and other poly(2,5 dialkoxyphenylenevinylenes) with at least one of the alkoxy groups  
10 being a long chain solubilising alkoxy group, polyfluorenes and oligofluorenes, polyphenylenes and oligophenylenes, polyanthracenes and oligo anthracenes, ploythiophenes and oligothiophenes and substituted polyfluorenes.

12. An electroluminescent device as claimed in any one of claims 3 to 11 in which the  
15 electroluminescent compound is mixed with the hole transmitting material.

13. An electroluminescent device as claimed in any one of claims 3 to 12 in which there is a layer of an electron transmitting material between the cathode and the electroluminescent compound layer.

20

14. An electroluminescent device as claimed in claim 13 in which the electron transmitting material is a metal quinolate.

15. An electroluminescent device as claimed in claim 14 in which the metal  
25 quinolate is an aluminium quinolate or lithium quinolate.

16. An electroluminescent device as claimed in claim 15 in which the electron transmitting material is of formula  $M_x(DBM)_n$  where  $M_x$  is a metal and DBM is

- 16 -

dibenzoyl methane and n is the valency of Mx or there is a Schiff base in place of the DBM.

17. An electroluminescent device as claimed in claim 13 in which the electron  
5 transmitting material is a cyano anthracene such as 9,10 dicyano anthracene, a polystyrene sulphonate or a compound of formulae shown in figure 1 or 2 of the drawings.

18. An electroluminescent device as claimed in any one of claims 13 to 17 in which  
10 the electron transmitting material is mixed with the electroluminescent compound.

19. An electroluminescent device as claimed in any one of the claims 3 to 18 in which the first electrode is a transparent electricity conducting glass electrode.

15 20. An electroluminescent device as claimed in any one of the claims 3 to 20 in which the second electrode is selected from aluminium, barium, rare earth metals, transition metals, calcium, lithium, magnesium and alloys thereof and silver/magnesium alloys.

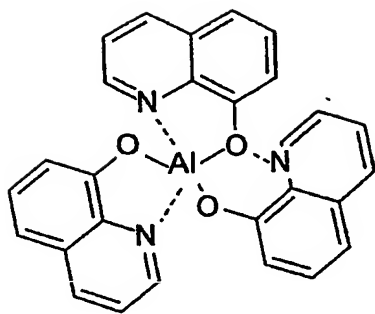
20 21. An electroluminescent device as claimed in any one of the claims 3 to 20 in which the second electrode is selected from lithium fluoride and other alkali metal fluorides rare earth metal fluorides by having a metal fluoride layer formed on a metal.

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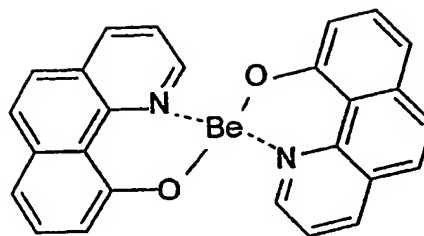
- 17 -

# Abstract

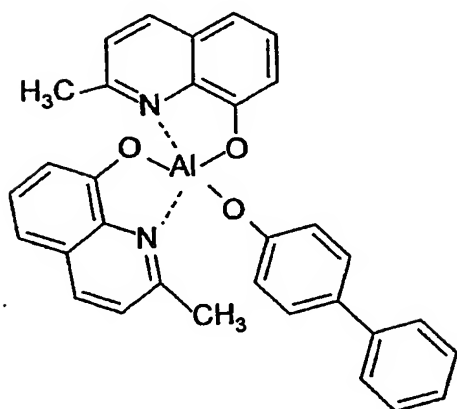
Novel boron complexes are blue emissive electroluminescent compounds.



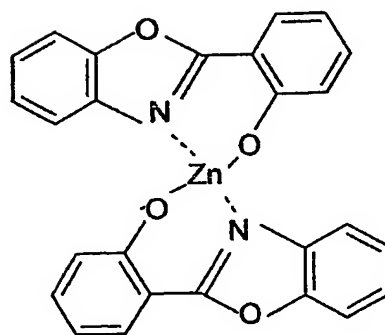
Alq



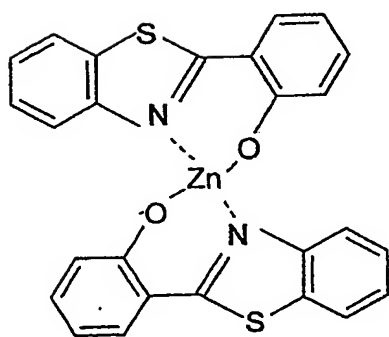
Bebq



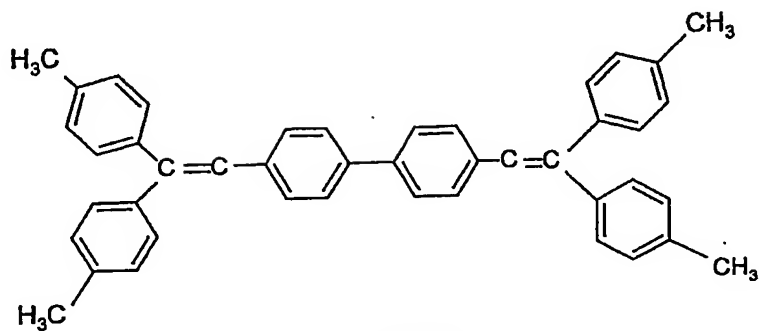
BAiq1



ZnPBO



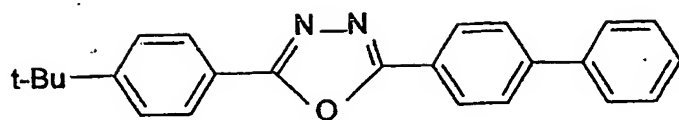
ZnPBT



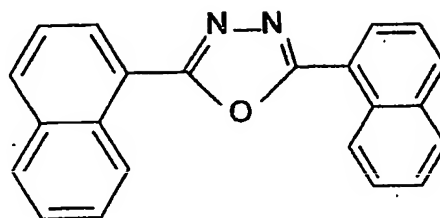
DTVb1

Fig. 1

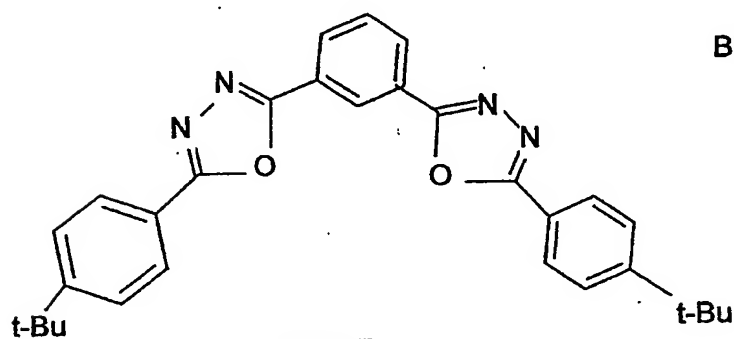
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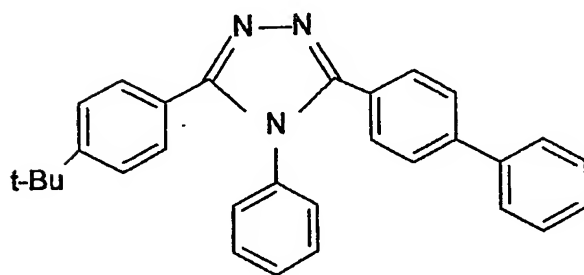
t-Bu-PBD



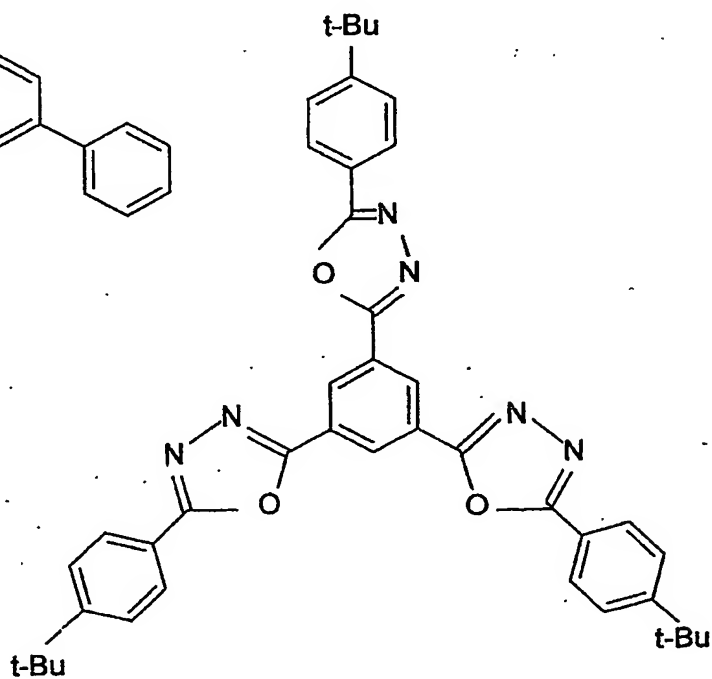
BND



OXD-7



TAZ



OXD-Star

Fig. 2

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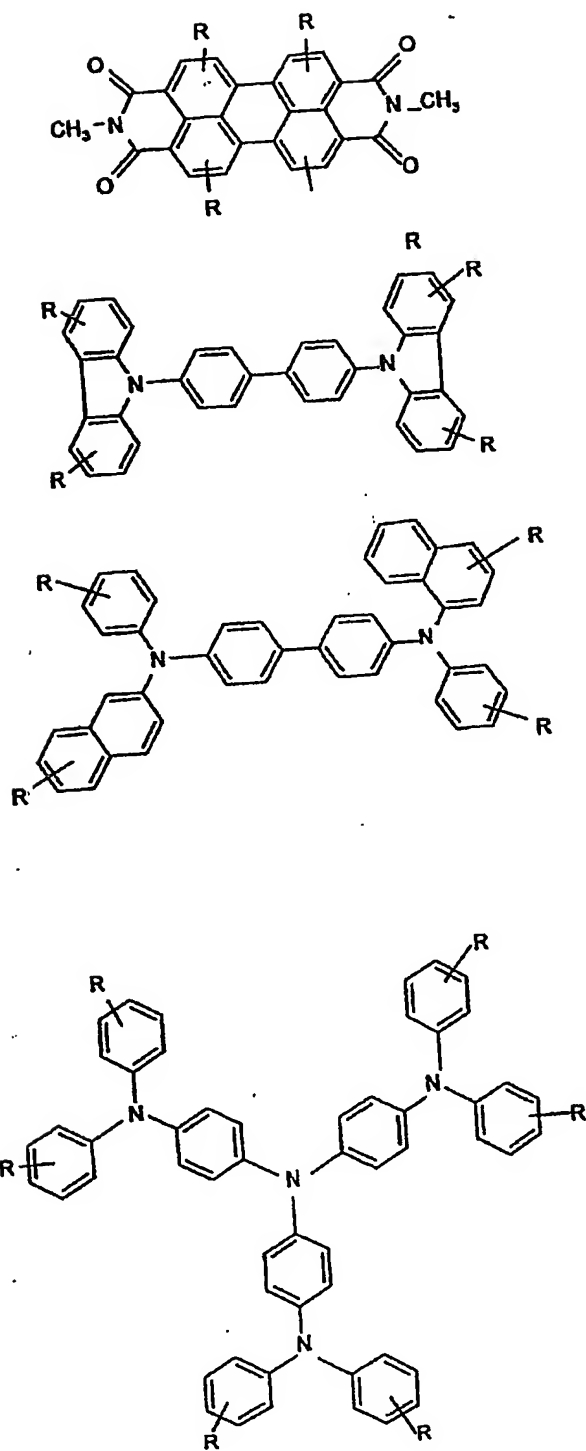


Fig. 3

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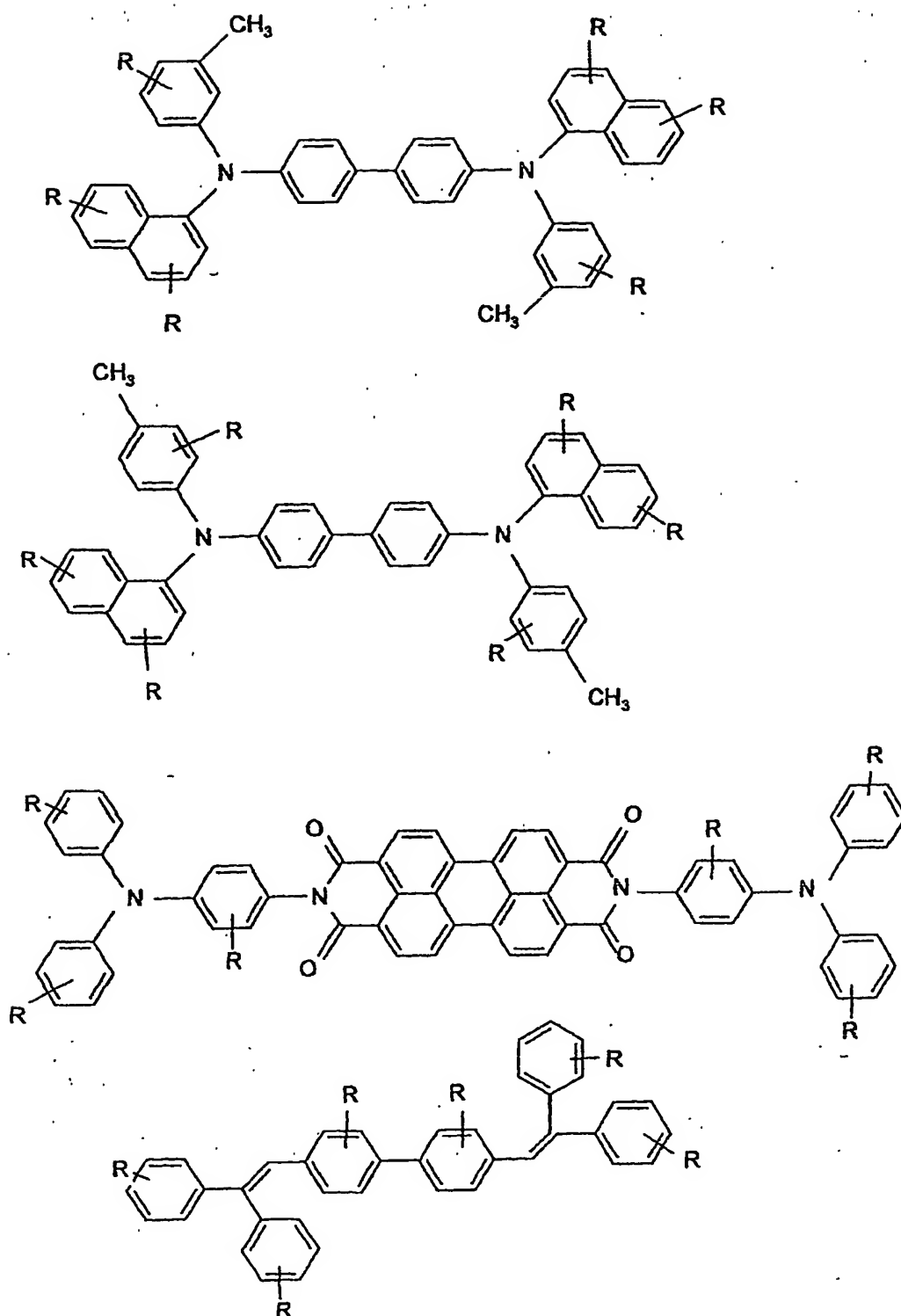


Fig. 4



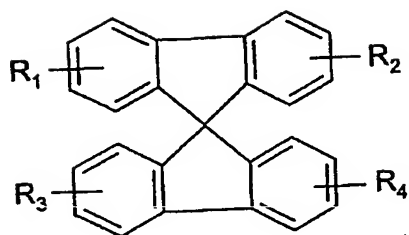


Fig. 14a

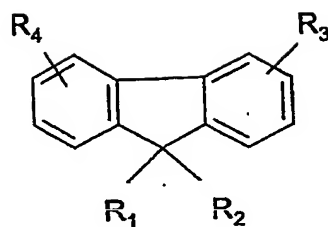
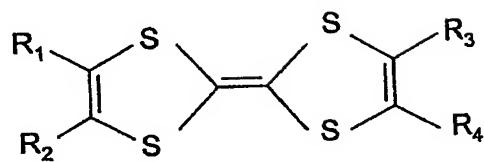


Fig. 14b



or

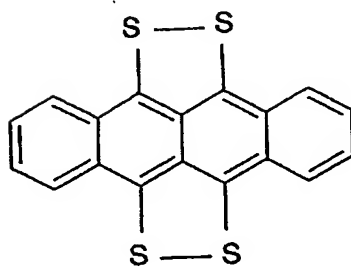
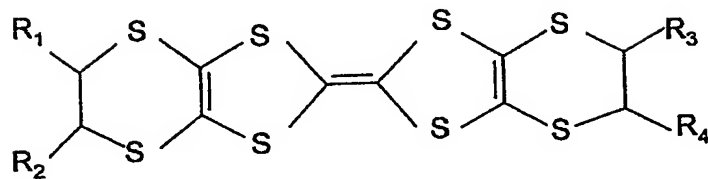


Fig. 5

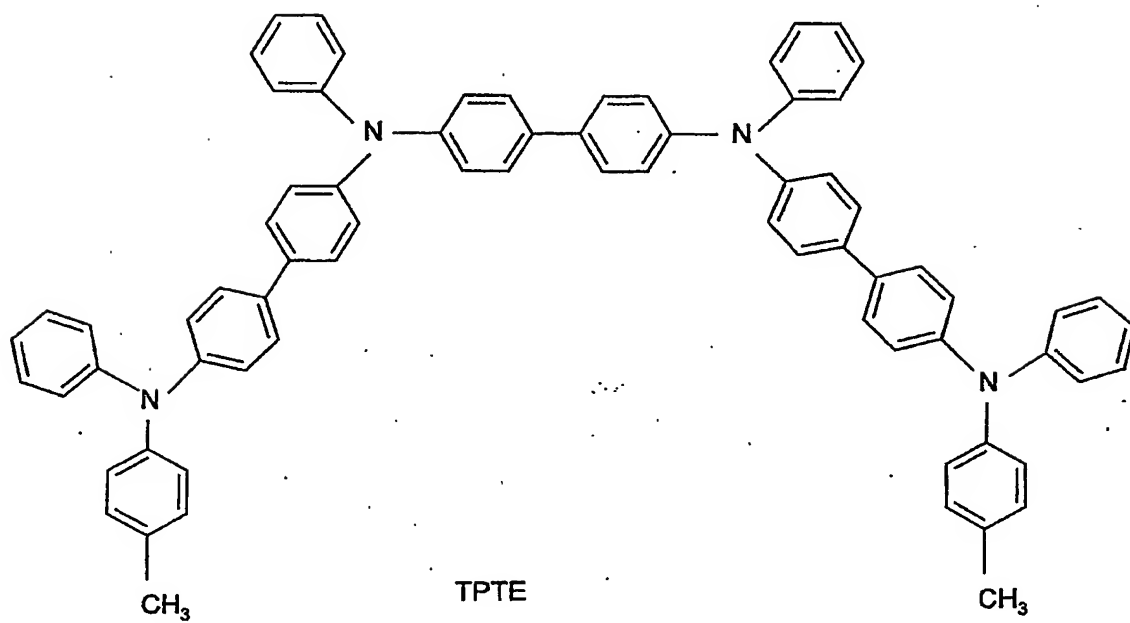
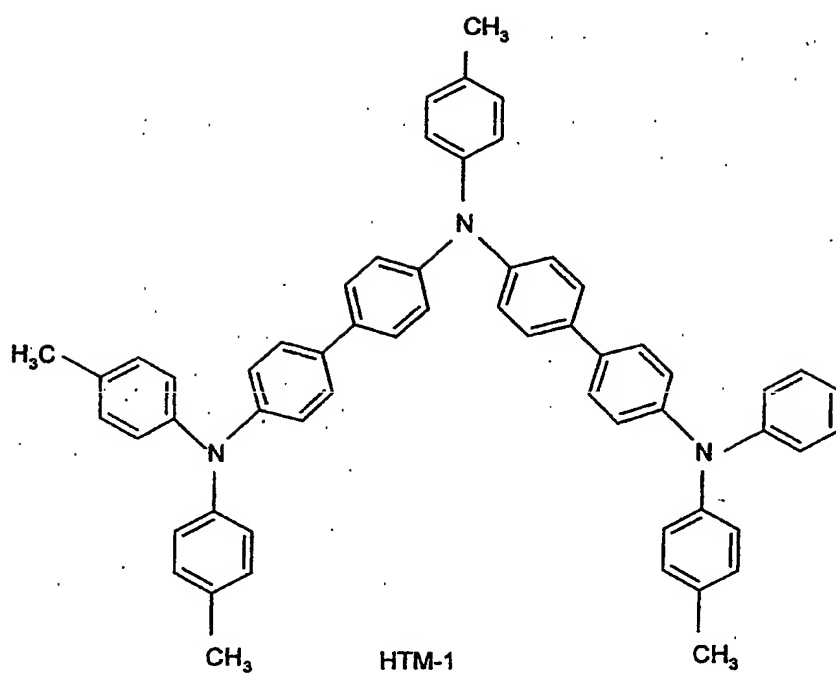
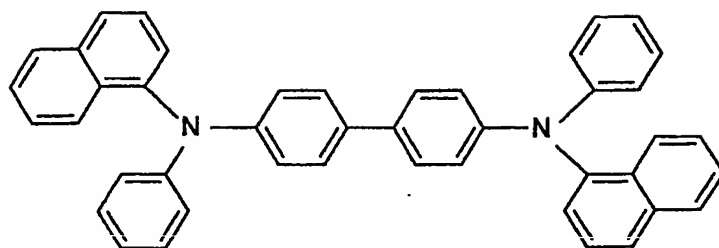
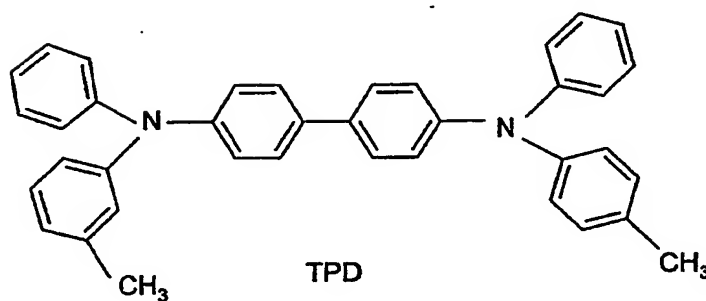


Fig. 6

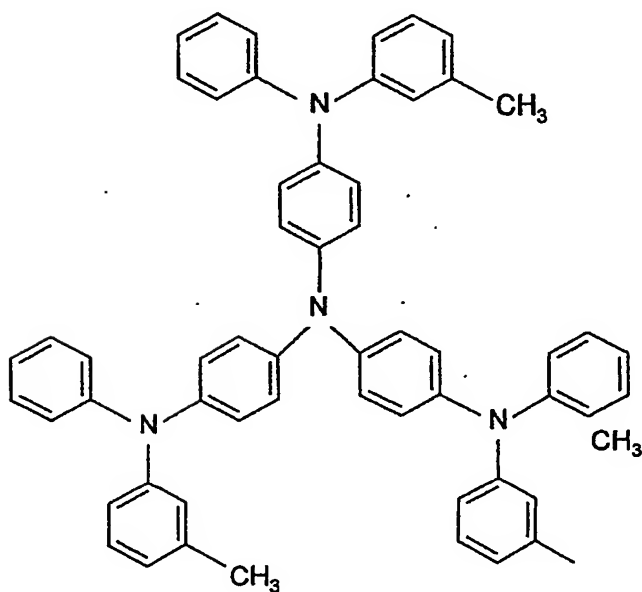
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$\alpha$ -NPB



TPD

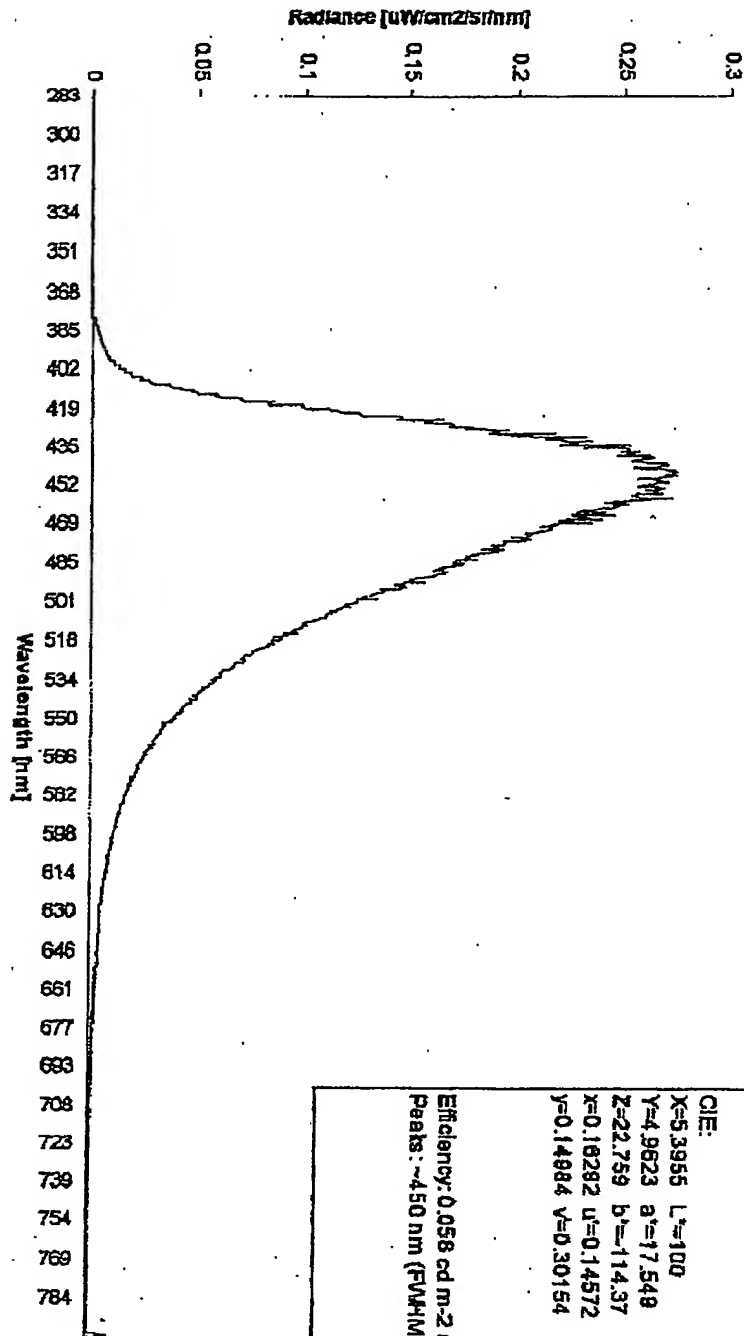


mTADATA

Fig. 7

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SK 11/11/02



Emission Spectra

CIE:  
 $X=5.3955$   $L^*=100$   
 $Y=4.9623$   $a^*=17.549$   
 $Z=22.759$   $b^*=114.37$   
 $x=0.18282$   $u=0.14572$   
 $y=0.14884$   $v=0.30154$

Efficiency: 0.056 cd m<sup>-2</sup> W<sup>-1</sup>  
 Peaks: ~450 nm (FWHM: ~80 nm)

Fig. 8

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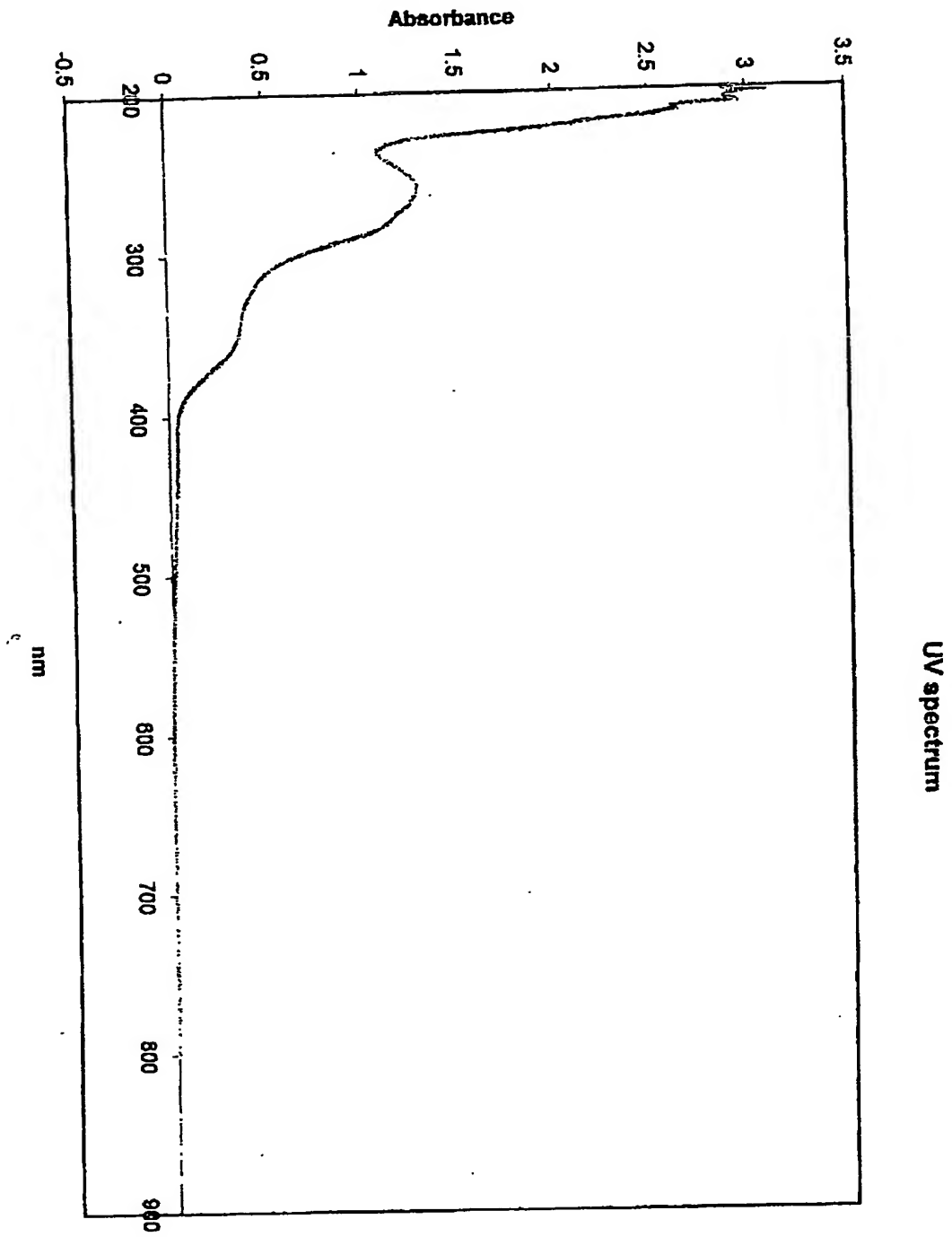


Fig. 9

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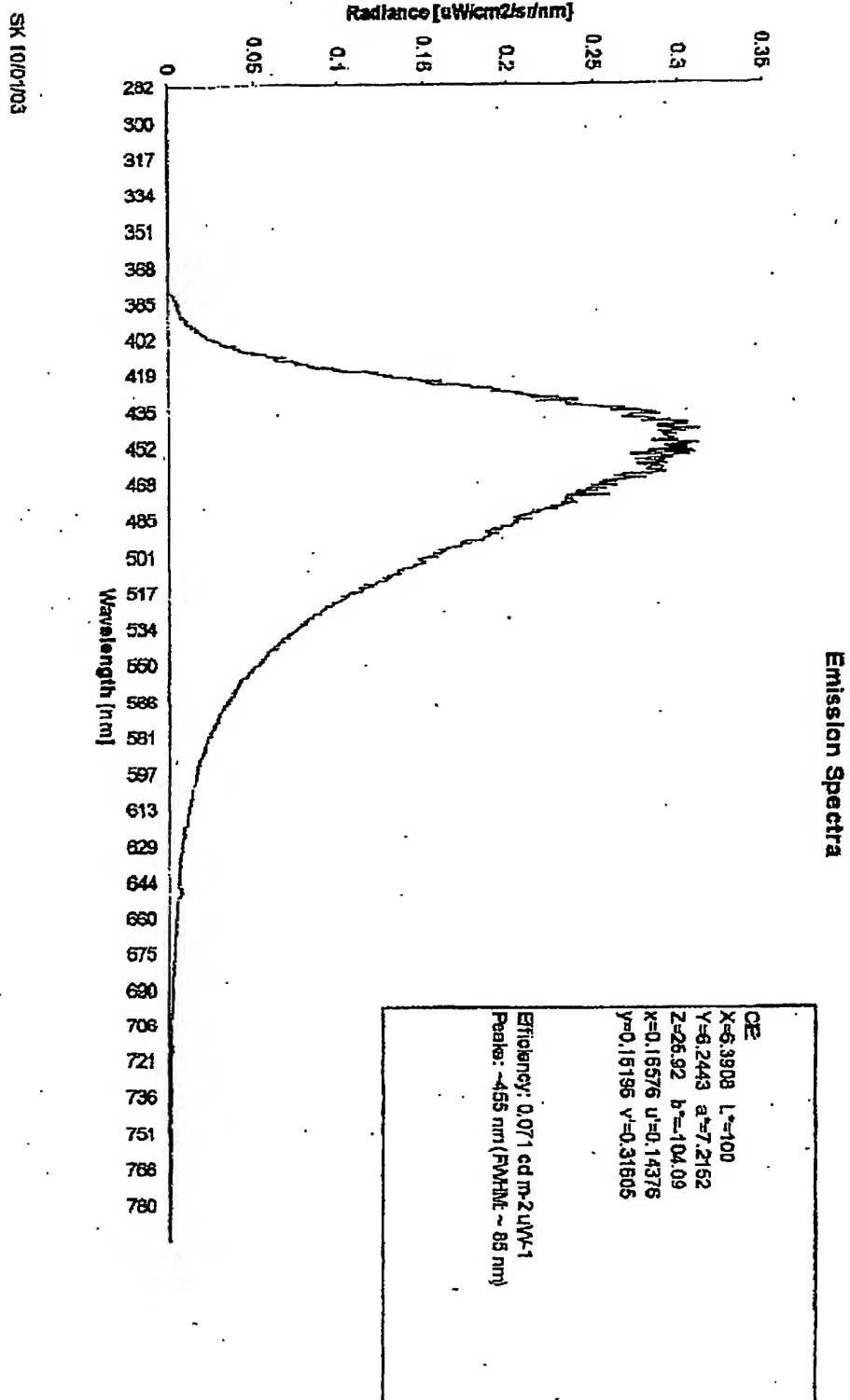


Fig. 10

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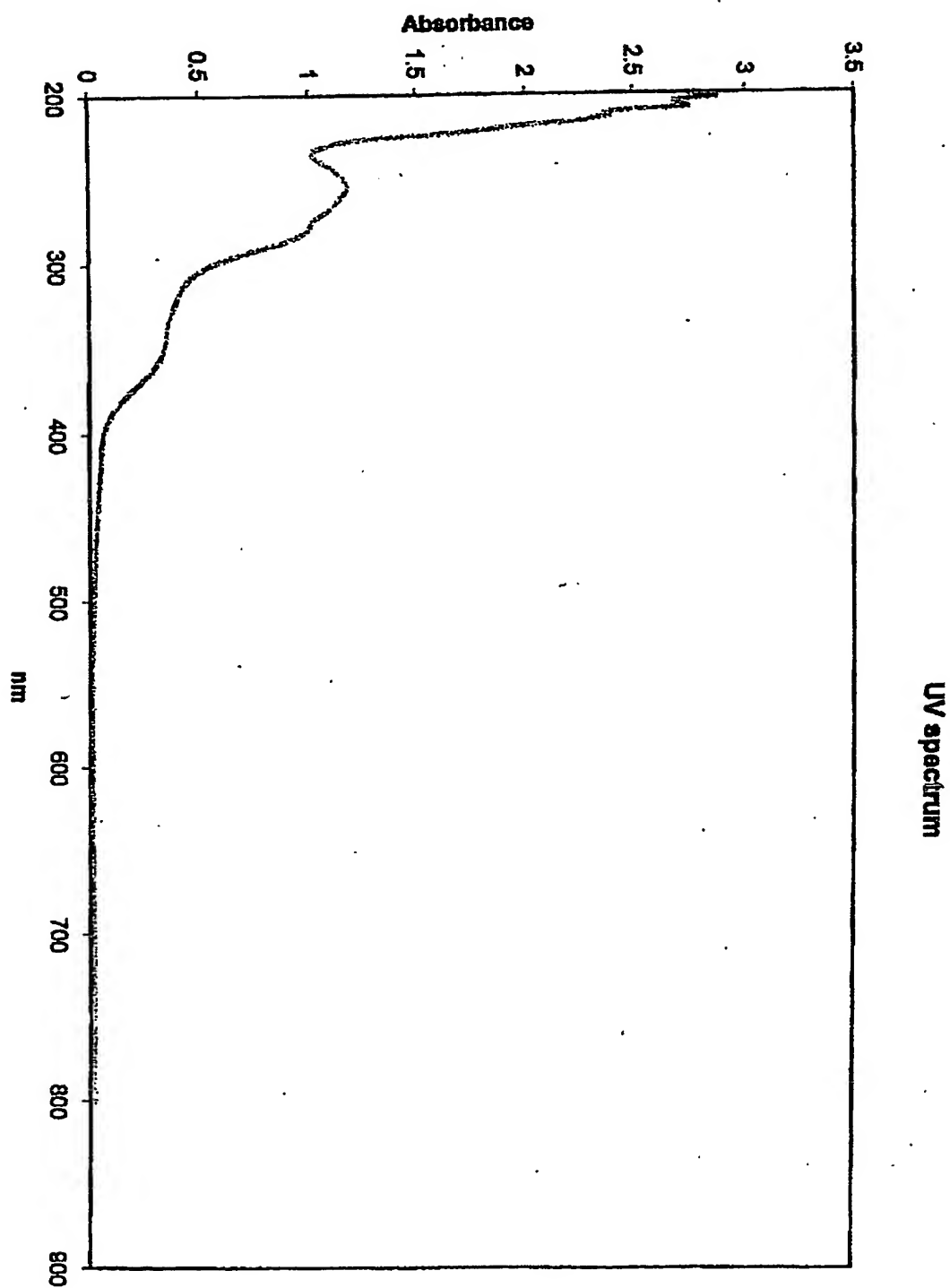


Fig. 11

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